INTERLABORATORY STUDY 91-3

PHENOLICS (4AAP) IN REAGENT WATER AND STP EFFLUENT

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INTERLABORATORY STUDY 91-3
PHENOLICS (4AAP) IN REAGENT WATER AND STP EFFLUENT

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1. SUMMARY AND CONCLUSIONS

This is the second interlaboratory study initiated, as part of an ongoing program of the Quality Management Office (QMO), Laboratory Services Branch (LSB) of the Ontario Ministry of the Environment (MOE) to evaluate the ability to measure phenolics in reagent water and STP effluent.

The previous MOE study (89-6) was confined to reagent water only and the findings were published elsewhere[1]. About two thirds of the participants in that study were flagged as not meeting the requirements of 'acceptable performance' of that study and a follow-up study was recommended. The term 'acceptable' throughout these studies is based on a factor times the estimated average repeatability of the participants.

Forty laboratories initially agreed to participate in the present study. Two laboratories failed to report results after receiving the samples.

Three samples in each of the two matrices, namely reagent water and STP effluent, were prepared by the QMO and distributed to the participants. The participants were requested to analyze using 4-AAP method specified in the MISA (Municipal and Industrial Strategy for Abatement) regulations[2].

The results as they were received were entered on to a Lotus 123® spreadsheet. A hard-copy of the electronic spread sheet was sent to all participants for verification. This enabled one laboratory to identify immediately a problem with their computer algorithms.

The performance of the participants was evaluated using King-Selliah[3,4] graphical procedure.

A comparative summary of findings of the two studies (in reagent water samples) is presented in Table 1.1

The findings of these two studies are essentially similar. The average recovery and the repeatability estimates of both studies are not significantly different. About one third of the participants in both studies demonstrated acceptable performance. Thus it may be concluded that about one third of the laboratories are capable of producing valid analytical data in simple matrices such as reagent water.

Eighteen laboratories participated in both studies. Among these, 14 laboratories showed no change in performance. This includes 4 laboratories that were assessed as acceptable performers in both studies. Ten laboratories were flagged in both studies. Three who were 'acceptable' in the previous study were flagged in the present study. One laboratory that was flagged in the previous study demonstrated acceptable performance in this study.

Preliminary scrutiny of the effluent sample data showed poor and highly variable recovery. Hence, only laboratories that were within control limits in reagent water were chosen for effluent sample evaluation. These laboratories while showing acceptable precision, reported results, on average, about 50 % of what was expected. The cause of this apparent 'under-recovery' cannot be identified at this time. A stability study of phenolic compounds in common effluents is being initiated.

'MISA' regulation method detection limit (RMDL) for 'phenolics by 4-AAP' is 2 µg/L. This requires laboratories to achieve a standard deviation (ie repeatability) of 0.7 µg/L or better. Laboratories whose reading increment are 0.5 µg/L or less are most likely to achieve this repeatability. Only about half the participants of this study demonstrated the capability of producing such precise data.

A noteworthy improvement in the present study is that a greater number of laboratories in the present study reported results containing 3 or more digits. This is particularly important when evaluating results that start with a 'one' (eg. 1.73). Three or more digits in an analytical result can be valuable to the data user as it will have a positive impact on bias estimation, trend monitoring and other calculations using laboratory analytical data.

TABLE 1.1

SUMMARY OF STUDY FINDINGS -
(Reagent water samples only)

	STUDY 91-3	STUDY 89-6
# of labs received samples	40	30
# of labs reported results	38	30
Acceptable performance (within warning limits)	12 (31.6%)	10 (33%)
Exceeded warning limits but within control limits	4 (10.5%)	4 (13%)
Out of control/Erratic	7 (18.4%)	4 (13%)
Low bias	9 (23.6%)	9 (30%)
High bias	6 (15.8%)	3 (10%)
Slope dependant bias	4 (10.5%)	3 (10%)
Intercept dependant bias	3 (7.9%)	6 (20%)
Repeatability (S_w)	.89 µg/L	.86 µg/L

2. TECHNICAL OUTLINE

2.1 SAMPLE PREPARATION

2.1.1 REAGENTS

i) Deionized Distilled Water (DDW)

ii) Homogenized STP Effluent

This effluent was collected from the Lakeview Sewage Treatment Plant on September 9, 1991. The effluent was thoroughly mixed and aerated for one week to remove any residual chlorine. This would also reduce the levels of phenolics in the original sample.

iii) MOE Phenol Standard, Lot# PHEN01 (10 mg/L)

iv) Concentrated Sulfuric Acid (Fisher- ACS grade)

2.1.2 PROCEDURE

30 L of each type of sample (listed in Table 2.1) was prepared. In every case 18 L (3 x 6L) of the matrix was quantitatively transferred to a clean stainless steel tank. 5 mL of concentrated Sulfuric Acid was added to the contents of the tank. A fourth portion of 6L matrix containing appropriate amounts of Phenol Standard (Table 2.1) was added to the tank. This was followed by the last 6L portion of the matrix. The contents of the tank were mixed thoroughly for 15 minutes using a mechanical stirrer.

With the aid of a peristaltic pump approximately 250 mL of this bulk solution was immediately dispensed into appropriately labelled special 250 mL 'phenol' bottles containing about 2mL of 50% Sulfuric Acid as preservative.

TABLE 2.1
Spiking chart for sample preparation

SAMPLE	SPIKING STANDARD	MATRIX
D1	None	Reagent water (DDW)
D2	60 mL	Reagent water (DDW)
D3	120 mL	Reagent water (DDW)
E1	None	STP Effluent
E2	60 mL	STP Effluent
E3	120 mL	STP Effluent

2.2 DISTRIBUTION

Prior to sample preparation, the participating laboratories received a letter of notification. The laboratories confirmed their willingness to participate in this study by letter or telephone. Each participant was assigned a laboratory identification code. These codes are different from those assigned in the previous MOE study. A list of participating laboratories is included in the Appendix (section 5.III).

A total of six samples were packaged in cardboard boxes and were shipped to most participants via Purolator courier on October 22, 1991. A few samples were hand delivered. No sample losses in transit were reported. Included with the samples were result reporting forms.

2.3 DATA HANDLING

The majority of the laboratories utilized the result reporting forms to submit their findings to the QMO. All data were manually entered by laboratory code into an electronic spreadsheet.

Tables of results were sent to all participants for verification on December 17, 1991. The participants were requested to report any transcriptional errors before January 10, 1992. One laboratory reported calculation errors on all their samples and re-submitted corrected results. Another laboratory reported a transcriptional error on their part in one of their results. In both cases the database was corrected accordingly. One laboratory identified problems with their computer algorithms and requested that their data be withdrawn from the evaluation. Their data was not withdrawn as it would have been automatically eliminated as outlier in the evaluation process.

3. RESULTS AND DISCUSSIONS

Forty laboratories received a total of six samples consisting of an unspiked sample, a low spike sample and a high spike sample in each of the two matrices. Two laboratories did not report any results.

The data was evaluated by the King-Selliah[3,4] graphical procedure. An outline of this automated procedure is given in the Appendix (5.I.1).

Based on the standard deviations of the 'selected laboratories' (i.e. after rejecting outliers), the evaluation is performed either on absolute scale (concentration units) or on relative scale (as percentage of target or median). The former will result in a rectangular graph and the latter will result in a square graph. In this study the evaluation was performed on absolute scale.

Warning and control limits have been used as the basis to flag laboratories that did not demonstrate acceptable repeatability or reproducibility. The term 'acceptable' throughout this evaluation is based on an estimate of average repeatability of all participants (excluding those too far from the 45 degree axis).

The K-S procedure allows data to be evaluated with respect to study median or expected value. In this instance the targets were known and hence the data was evaluated with respect to expected values.

Preliminary evaluation of reagent water data resulted in a repeatability estimate that was not only significantly different from that of the previous study, but also much higher than that imposed by the 'MISA' regulation method detection limit (RMDL) criteria. (RMDL is usually set at 3 times the acceptable repeatability). A closer look at the distribution of PD data (perpendicular distance to the 45 degree line used to estimate repeatability [3,4,5]) showed that there were at least two populations and the median PD did not represent any central tendency. This would indicate that there are two or more levels of 'repeatability performance' present among the participants. Thus the K-S procedure was permanently revised to include an iterative process that will result in a stable median representing a more precise group of participants.

Reagent water samples.

As expected, the majority of the laboratories reported 'not detected' (or less than detection limits) for the unspiked reagent water sample. Hence, the results of this sample were not used in the graphical evaluation of the spiked portion.

Thirty eight laboratories reported results in this study. Among these eighteen participated in the previous study[1] (MOE 89-6). Comparative summaries of findings of these two studies are presented in Tables 1.0, 3.1 & 3.2. Table 3.1 is the statistical summary of both studies. The repeatability (S_w) indicated in this table represents the average within-laboratory standard deviation estimated based on the results of each study as described in the Appendix (section 5.I.1).

TABLE 3.1

STATISTICAL SUMMARY

	STUDY 91-3		STUDY 89-6*	
SAMPLE	D2	D3	PHN 2	PHN 3
Target (µg/L)	20	40	10	40
# of digits reported: 3 or more	19 (50%)	20 (52.6%)	4 (13%)	7 (23%)
# of digits reported: 2	18 (47.4%)	18 (47.4%)	10 (33%)	19 (49%)
# of digits reported: 1	1 (2.6%)	-	16 (53%)	4 (13%)
ALL LABORATORIES				
# of Labs	38	38	30	30
Mean(µg/L)	18.3	48.2	9.7	50
Median(µg/L)	19.0	40	8.9	39
AFTER ELIMINATING OUTLIERS				
# of Labs	26	29	20	23
Mean(µg/L)	18.3	39.2	8.8	39
Median(µg/L)	18.8	40	9.9	39
S(µg/L)	1.878	3.245	1.9	2.0
Average recovery (median/target x 100)	94%	100%	99%	97.5%
Repeatability S_w (µg/L)	.89		0.86	

* RESULTS OF STUDY 89-6 WERE REPORTED IN MG/L. THEY WERE CONVERTED TO µG/L IN THE ABOVE TABLE.

Table 3.2 presents the outcome of evaluations of individual laboratories in both studies. Evaluation outcomes of those laboratories that participated in both studies are presented on the same row.

The key to this table is as follows:

A	Acceptable performance
ER	Both results erratic
H	Biased high
He	Biased high and/or erratic
Hi	Biased high, possible intercept problem
HI	Biased high, probable intercept problem
Hs	Biased high, possible slope problem
HS	Biased high, probable slope problem
Le	Biased low and/or erratic
Li	Biased low, possible intercept problem
LI	Biased low, probable intercept problem
Ls	Biased low, possible slope problem
LS	Biased low, probable slope problem
OC	Out of control-one result erratic
WAI	Warning: slight imprecision
WLS	Warning: Biased low, probable slope problem
WLi	Warning: Biased low, possible intercept problem
WLI	Warning: Biased low, probable intercept problem
WOC	Warning: Out of control-one result erratic

TABLE 3.2

SUMMARY OF LABORATORY PERFORMANCE

STUDY 91-3		STUDY 89-6
LAB CODE	PERFORMANCE CODE	PERFORMANCE CODE
9002	A	A
9003	A	
9004	ER	
9005	A	A
9006	A	
9010	LS	OC
9011	ER	Li
9012	LS	OC
9013	OC	
9014	OC	
9015	LS	
9016	A	A
9017	WLi	
9018	WLI	LI
9019	A	A
9020	A	
9021	Le	Le
9023	OC	
9024	Le	HI
9025	Le	Ls
9026	He	ER
9027	A	
9028	A	
9029	Le	OC
9030	WLI	
9031	Hi	A
9033	LI	A
9034	OC	A
9035	He	
9037	WAI	
9038	He	
9039	H	
9040	A	
9041	A	
9043	LI	LS
9044	A	LI
9047	Hs	
9048	ER	

Figure 3.1 is the graphical presentation of the findings of the present study. Each point represents a pair of results from each participant. Laboratories that have exceeded the warning limits have been identified. A detailed explanation of the two sample plots are given in the Appendix (section 5.I).

12 laboratories (31.6%) in the present study demonstrated acceptable performance. These laboratories can be considered as capable of producing valid analytical data in simple matrices such as reagent water. Another 4 (total of 16; 42%) produced results that were within control limits.

Concentration dependant biases (slope biases) appear to be the major problem for four laboratories. Such biases are caused by either inaccurate standards or inadequate calibration procedure. Use of certified reference standards or use of the same source of 'quality' external standards by all laboratories will probably improve the data comparability.

Three laboratories demonstrated intercept dependant bias. Such biases are caused by inappropriate base-line and/or background and/or blank correction. In automated continuous flow systems the 'wash' water used between samples may be contaminated with low levels of phenols.

Seven laboratories in this study were flagged as erratic or out of control. These seven laboratories must achieve greater control over the entire analytical system before a diagnosis of biases is possible.

The initial evaluation of the data yielded a high estimate of repeatability. 'MISA' regulation method detection limit (RMDL) for 'phenolics by 4-AAP' is 2 µg/L. This requires laboratories to achieve a standard deviation (ie repeatability) of 0.7 µg/L or better. Such precision is most likely achieved by those who are able to read to the nearest 0.5 µg/L. Only about half the participants of this study demonstrated this capability of producing such precise data. A revision to the K-S procedure for determining median PD was required.

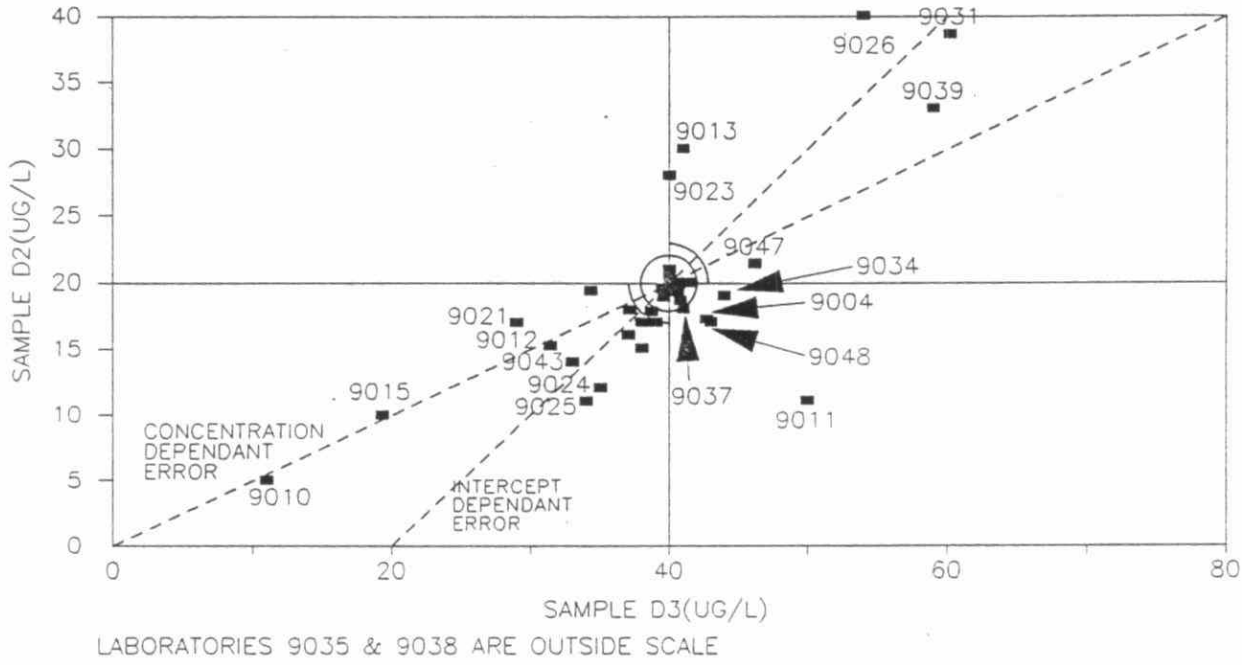
A comparative summary of findings of the two studies MOE 89-6 and MOE 91-3 (in reagent water samples) is presented in Table 1.1 (page 3).

Based on the revised procedure for determining median PD, the findings of these two studies are essentially similar. The average recovery and the repeatability estimates of both studies are not significantly different. About one third of the participants in both studies demonstrated acceptable performance. Thus it may be concluded that about one third of the laboratories are capable of producing valid analytical data in simple matrices such as reagent water.

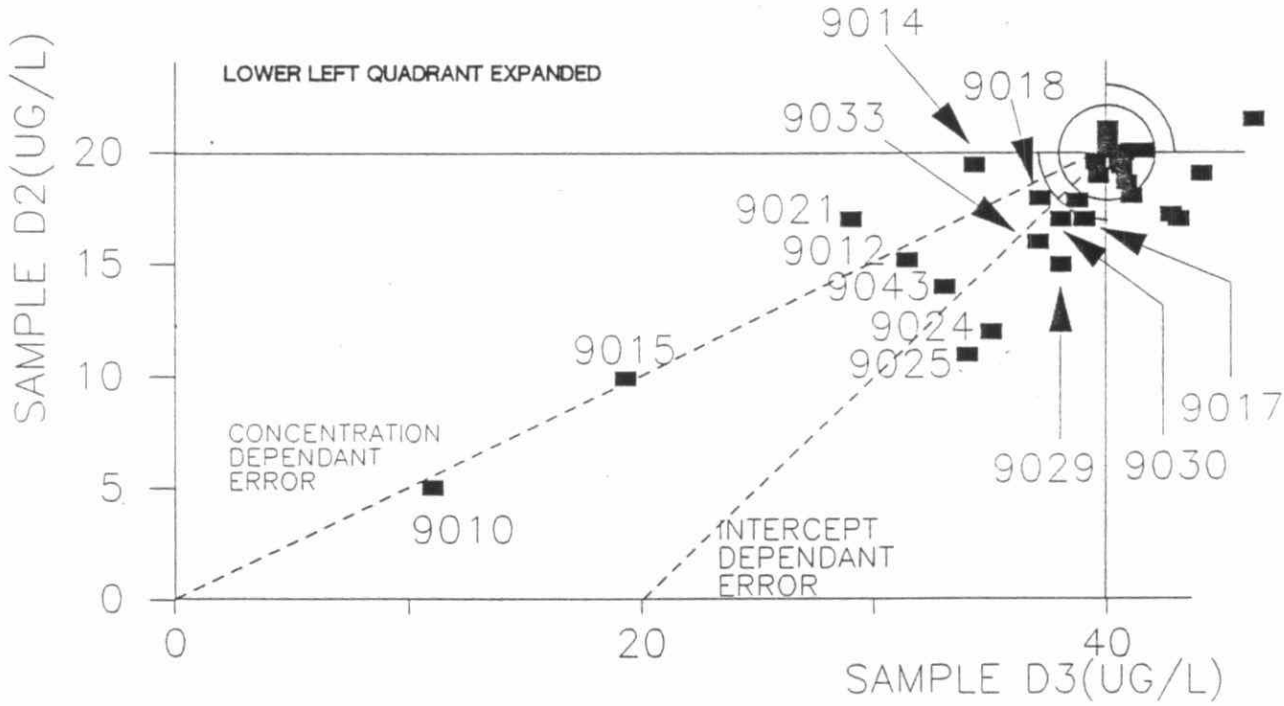
FIGURE 3.1

STUDY 91-3

SAMPLE D2 VS SAMPLE D3



LABORATORIES 9035 & 9038 ARE OUTSIDE SCALE



Eighteen laboratories participated in both studies. Among these, 14 laboratories showed no change in performance. This includes 4 laboratories that were assessed as acceptable performers in both studies. Ten laboratories were flagged in both studies as not producing acceptable results. Three who were 'acceptable' in the previous study were flagged in the present study. One laboratory that was flagged in the previous study demonstrated acceptable performance in this study.

In the present study fewer laboratories exhibited problems with 'blank' correction. This is a noticeable improvement.

About 50% of the participants in the present study reported three or more digits. This is an improvement from the last study. It is recognized that unnecessary truncation of results introduces a bias which certainly affects the evaluation of laboratory performance, and which can affect the evaluation of spatial or temporal trends in environmental databases. Laboratories reporting more digits tend to demonstrate better control over blank/baseline effects.

STP Effluent Samples.

Preliminary examination of effluent data indicated that the average recovery for the spiked effluent was about 50% of the expected. Furthermore, the between laboratory variability of each spiked effluent sample even after excluding outliers was much greater than the corresponding reagent water sample.

Because of the high variability of the effluent data only laboratories that were within control limits in reagent water samples were chosen for further evaluation. Reagent water samples are essentially spiked standards and the rationale for limiting the evaluation to selected participants is that laboratories must establish proficiency in analyzing standards before they could be evaluated for 'real samples'.

Figure 3.2 presents the outcome of the evaluation. The solid diagonal lines representing the two types of biases (slope dependant and intercept dependant) are identified. The two dotted lines parallel to the slope error lines are the precision limits about the slope error line. The precision limits are set at +/- 2 times repeatability (S_w estimated from the reagent water data). The point representing the two targets are identified by a cross within a small circle. This is also the point where the two error lines intersect.

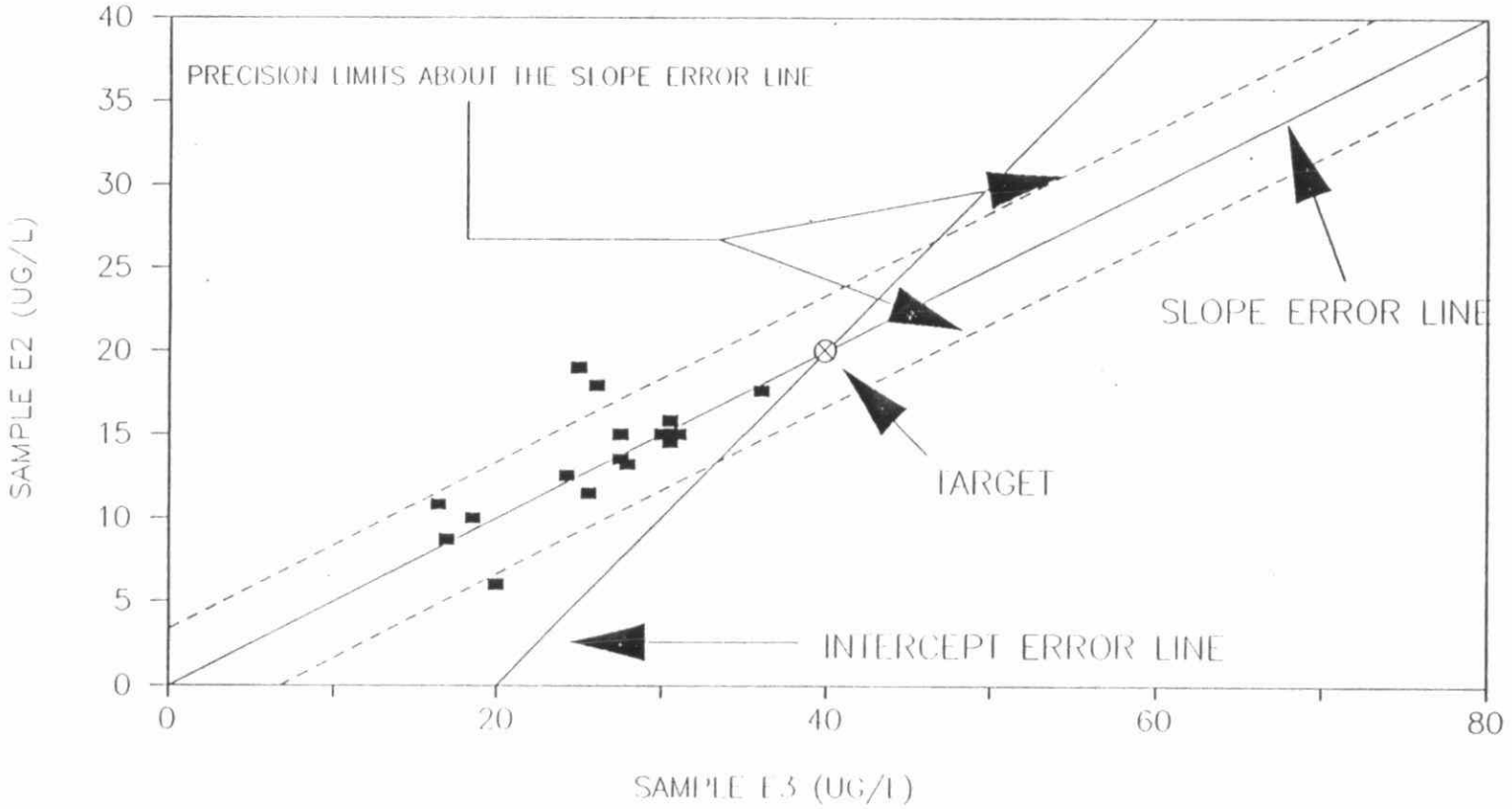
The majority of the selected laboratories showed acceptable precision in their effluent data, but reported considerably lower values than expected. This is reflected in the diagram as slope dependant biases.

FIGURE 3.2

STUDY 91-3

SAMPLE E2 VS SAMPLE E3 (SELECTED LABORATORIES ONLY)

(MATRIX: STP EFFLUENT)



Close scrutiny of the QMO records do not give any reasons to suspect the accuracy of spiking. The samples were prepared and stored, prior to transportation, under conditions stipulated in the 'MISA' regulations. Even though pilot studies indicated no loss of phenol content on storage of spiked effluent samples, this study seems to indicate that there was a degradation. At this stage it is not possible to say whether low and variable recovery is due to inadequacy in the method to handle complex matrices such as STP effluent or instability of 'phenol' in the effluent (Oxidation, Microbial action, conversion to less volatile chlorophenols etc.). A detailed study of stability profiles of phenolic compounds in a variety of STP and industrial effluents under various storage and transportation conditions is being initiated.

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5. APPENDIX

I. EVALUATION METHODOLOGY

Evaluation of the laboratory performance in this study was performed by the King-Selliah[3,4]. An outline of this automated spreadsheet- graphical procedure is given below.

I.1 Summary of The Two-Sample Performance Evaluation Procedure

1. <u>Sample distribution-data receipt</u>

   i) Split two samples of different concentrations among a number of laboratories for analysis/measurement using their current methodology.
   ii) Enter data on LOTUS 123® spreadsheet.
   iii) Calculate median (L_m,H_m), means and standard deviations for each sample.
   iv) Tabulate data and return to laboratory analyst for verification.
   v) Correct database if transcriptional errors were reported.

2. <u>High sample data evaluation:</u>

   i) reject all results which differ from the median (H_m) by more than 10%
   ii) calculate median(H), mean and standard deviation (S_h)
   iii) re-include data if within 3 times S_h
   iv) reiterate ii) and iii) until no further data is included
   v) calculate relative standard deviation of the final selected data (CV_h)

3. <u>Low sample data evaluation:</u>

   i) use 3 x CV_h x median(L_m) to exclude possible outliers
   ii) calculate median(L), mean and standard deviation (S_l)
   iii) reinclude data if within 3 times S_l
   iv) reiterate ii) and iii) until no further data is included.

4. <u>Sample data plot format:</u>

   i) examine the ratio of S_h/S_l and if:
   <2 use data as reported in <u>concentration</u> units
   otherwise convert to <u>% recovery</u> based on expected value if known (otherwise use median values (H,L))
   ii) prepare paired sample scatter diagrams of all data

5. **Performance criteria**

i) calculate perpendicular distance from each point to the two error lines (PD_{slope}, $PD_{intercept}$).
ii) determine the median (PD_{median}) of all perpendicular distances to the appropriate 45 degree line (intercept error line or slope error line for absolute or relative scale respectively)
iii) calculate the bias for each laboratory (ie the distance along the appropriate 45 degree line) and if:
<4.5 times PD_{median} select the PD to the 45 degree line
other wise select the lesser of the two PDs (PD_{slope} or $PD_{intercept}$)
iv) determine the median ($PD_{sel\text{-}med}$) of selected PDs .
v) exclude PD values(among previous selection) greater than 2.5 times the $PD_{(sel\text{-}med)}$.
vi) determine the median ($PD_{sel\text{-}med}$) of remaining PDs.
vii) Reiterate steps iv) to vi) until $PD_{(sel\text{-}med)}$ remains unchanged.

6. **Set Warning and Control limits**

i) determine the average of all selected PD values less than 2.5 times the $PD_{(sel\text{-}med)}$ and use this average to estimate the average repeatability S_w (see reference 5)
ii) set warning limits for repeatability = 2 times S_w
iii) set control limits for repeatability = 3 times S_w
iv) set warning limits for possible bias = 3 times S_w
v) set control limits for possible bias = 4.5 times S_w

6. **Code laboratory performance using automated program based on location**

i) in upper left or lower right quadrant (erratic)
ii) in lower left or upper right quadrant (biased low or high)
iii) on horizontal or vertical axis (out of control)
iv) on diagonal line through origin (slope or standard problems)
v) on diagonal line not through origin (intercept or blank problems)
vi) prepare performance assessment table

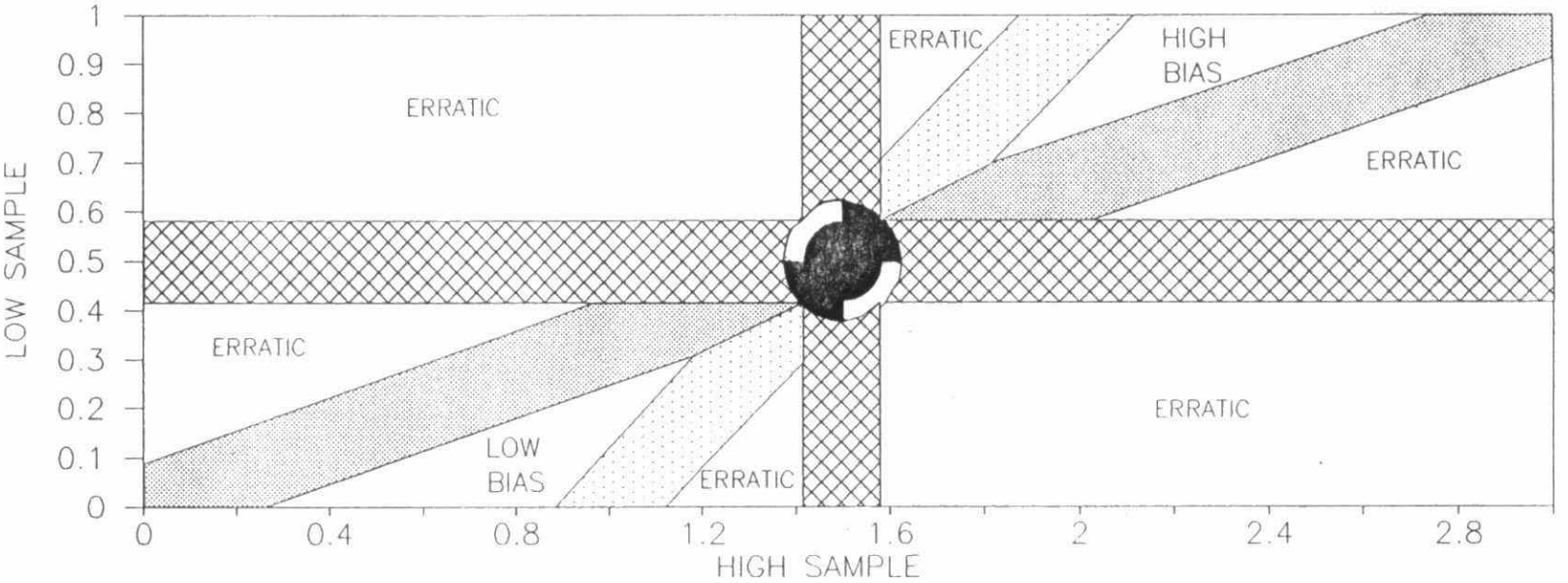
I.2 Limits for Maximum Interlaboratory Repeatability and Reproducibility

The average perpendicular distance (PD) from the bias lines represents the interlaboratory estimate of within laboratory repeatability. It is used to estimate S_w (see Appendix I.1). In some cases the distribution of PD's may suggest two or more levels of laboratory repeatability performance. In such a case the initial median may not represent the central tendency of the data. Therefore an iterative evaluation of the median is included to ensure a stable estimate of repeatability representing more precise group of participants.

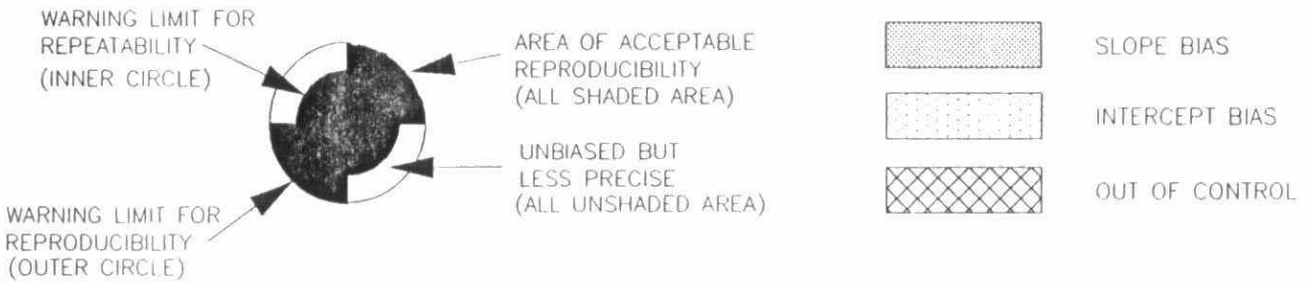
Warning limits and control limits for repeatability are set at $2S_w$ and $3S_w$, respectively. Note that the factors used are somewhat arbitrary but they represent approximately 95% and 99% confidence intervals.

Additional tolerance is required for the effect of variability in preparing and using standards on a day to day or among laboratory basis. But the overall estimate of reproducibility includes data from laboratories with excessive bias. In lieu of this, as a criterion for acceptable reproducibility. S_r is set at 1.5 S_w. Therefore the warning and control limits for reproducibility are set at $3S_w$ and $4.5S_w$ respectively. Based on the f-test a ratio $(S_r/S_w)^2$ exceeding 2.3 (i.e 1.5^2) would be considered significant with a risk of error of less than 10%, 5%, and 1% respectively for 10, 20, and 35 degrees of freedom. Results that exceed warning and control limits determined from this desired maximum interlaboratory (DMI) reproducibility (S_r) are deemed to be possibly or probably biased respectively.

FIGURE 4.0



KEY TO SHADED AREAS



I.3 Two Sample Plot Discussion

This graphical presentation enables all participants to visualize how they have performed compared to others. The assessment of a laboratory in this study is based on the location of its result on the graph. Figure 4.0 identifies the various regions in a typical graph associated with the different types of problems that might be experienced by the participants. Laboratories with controlled repeatability but showing various degrees of bias will appear in the lower left and upper right quadrants. The two circles drawn in this diagram represent the warning limits for repeatability (S_w) and reproducibility (S_r). Those points within the outer circle but in the upper left and lower right quadrants (not shaded in Figure 4.0) are unbiased but somewhat less precise. Those points within the circle but in the upper right and lower left quadrants are precise but acceptably biased. Thus the area of acceptable performance in this diagram has taken the shape of a keyhole.

In a typical graphical presentation such as Figure 3.1, the actual results of each laboratory constitute the points on the graph. The solid lines dividing the graph into four quadrants represent the median results of appropriate samples. The 'keyhole' shaped area of acceptable performance, described earlier, is the area de-limited by the inner circle and the outer arcs in the lower left and upper right quadrants. All laboratories that lie outside this area have exceeded the respective warning limits. The two dotted lines are drawn across the graph representing the slope (concentration) dependant error and the intercept (blank) dependant error. The laboratories that exhibit these types of biases will lie along these lines. All laboratories exceeding warning limits have been identified by their laboratory codes. These laboratories can readily see the nature of their particular problems.

II. RAW DATA

TABLE 4.1

LAB CODE	D1	D2	D3	E1	E2	E3
AMT. SPIKED	NONE	20	40	NONE	20	40
9002	0.6	19.9	40.7	3.3	17.6	36.0
9003	0.2	20.5	40.0	1.0	13.2	27.9
9004	1.16	17.2	42.7	6.30	7.39	20.9
9005	<1.0	20.0	41.0	1.0	14.5	30.5
9006	<MDL	18.9	39.6	1.3	10.9	22.4
9010	NR	5	11	1	3	6
9011	<2	11	50	7	<2	24
9012	<1.0	15.2	31.4	1.0	7.1	12.4
9013	5.1	30	41	12	21	19
9014	7.4	19.4	34.3	6.9	15.3	23.9
9015	6.40	9.94	19.3	2.66	7.47	7.97
9016	<1.0	19.5	39.5	<1.0	13.5	27.5
9017	<2	17	39	<2	10	23
9018	1.9	17.9	37.1	5.3	8.7	16.9
9019	<0.6	17.8	38.7	<0.6	6.0	19.9
9020	<1	21	40	3.5	15	27.5
9021	<2	17	29	4	10	17
9023	<1	28	40	6	15	27
9024	6	12	35	3	8	15
9025	<2	11	34	<2	6	12
9026	97	40	54	44	36	64
9027	1.3	19.5	40.5	3.5	15.8	30.5
9028	<2	20	41.5	13.5	19	25
9029	<2	15	38	3	13	24
9030	<1.9	17.0	38.0	<1.9	15.0	31.0
9031	21.1	38.6	60.2	27.4	33.0	42.4
9033	<1	16	37	4	17	22
9034	<DL	19	44	5	16	28
9035	35	70	70	<DL	20	35
9037	<1.5	18.0	41.0	3.5	10.0	18.5
9038	426	162	340	179	288	136
9039	<2	33	59	14	27	35
9040	<1	20	40	2	15	30
9041	<6	18.6	40.8	<6	17.9	26.1
9043	NIL	14	33	NIL	8.6	18
9044	1.1	19.3	40.6	3.4	10.8	16.4
9047	<1.0	21.4	46.2	3.8	14.0	22.6
9048	<2	17	43	9	12	25

III. LIST OF PARTICIPANTS

1 ASL ANALYTICAL SREVICE LABORATORIES LTD.

2 ATOMIC ENERGY OF CANADA LTD., CHALK RIVER, ONTARIO

3 ATOMIC ENERGY OF CANADA LTD., PINAWA, MANITOBA

4 BARRINGER LABS LTD.

5 BAS LABS LTD.

6 BONDAR CLEGG

7 CANTEST LTD.

8 CANVIRO ANALYTICAL LABORATORIES LTD.

9 CITY OF VANCOUVER

10 CLAYTON ENVIRONMENTAL CONSULTANTS

11 CONESTOGA ROVERS & ASSOC. LTD.

12 EAG ANALYTICAL SERVICES

13 ENVIROCLEAN

14 ENVIRONMENTAL PROTECTION LABORATORIES INC.

15 ESSO PETROLEUM CANADA

16 INCO LTD.

17 LAKEFIELD RESEARCH

18 MICROBE INC.

19 NOVACOR CHEMICALS (CANADA) LTD.

20 NOVALAB

21 ONTARIO HYDRO

22 ONTARIO MINISTRY OF THE ENVIRONMENT, LONDON

23 ONTARIO MINISTRY OF THE ENVIRONMENT, REXDALE

24 ONTARIO MINISTRY OF THE ENVIRONMENT, REXDALE

25 PARACEL LABORATORIES. LTD

26 PETRO CANADA LTD.,MISSISSAUGA

27 PETRO CANADA LTD.,OAKVILLE

28 POLLUTECH LTD.

29 PROCTOR AND REDFERN

30 REGIONAL MUNICIPALITY OF WATERLOO

31 SHELL CANADA PRODUCTS LTD.

32 SUNOCO - SARNIA REFINERY

33 TECHNICAL SERVICE LABORATORIES

34 WALKER LABORATORIES

35 WASTEWATER TECHNOLOGY CENTRE

36 XRAL ENVIRONMENTAL

37 ZENON ENVIRONMENTAL INC., ONT.

38 ZENON ENVIRONMENTAL INC.,B.C.